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HYDROGEN EMBRITTLEMENT OF NICKEL: MODELING OF THE  
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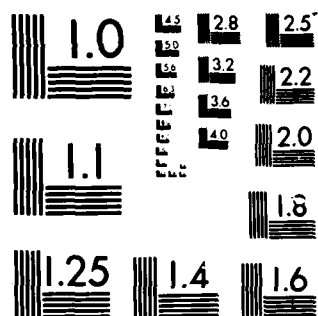
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HYDROGEN EMBRITTLEMENT OF NICKEL: MODELING OF THE EFFECT OF  
DIFFUSIVE SEGREGATION OF HYDROGEN ON INTERGRANULAR FRACTURE

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Summary

Grain boundary fracture of hydrogen-charged nickel was studied under test conditions where hydrogen is essentially immobile. Prior to testing, hydrogen was allowed to diffuse during aging treatments. Experimental results show that the transition in the fracture mode from ductile rupture to intergranular is strongly dependent on aging temperature and time as well as on initial bulk hydrogen concentration. Analytical modeling of these dependencies using established thermodynamic and kinetic relationships, indicates that grain boundary hydrogen concentration controls the fracture mode of hydrogen-charged nickel at low temperatures.



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## Introduction

Previous studies have shown that various concentrations of hydrogen in solid solution will considerably reduce the ductility of polycrystalline nickel deformed in tension over a wide range of test temperatures and strain rates (1-6). This reduction in ductility is caused by a change in fracture mode from ductile microvoid coalescence to an intergranular fracture mode. The Ni-H alloy retains significant, albeit reduced, ductility--in essence, premature failure occurs because of the intervention of grain boundary fracture. Although embrittlement of hydrogen-charged nickel has been studied extensively, this phenomenon is not well understood.

Intergranular fracture of nickel is, in most cases, promoted by the concentration of impurities at grain boundaries which occur in trace amounts in the bulk (7). For example, direct observation of sulfur segregation at nickel grain boundaries by auger electron spectroscopy (AES) has been used to correlate sulfur distributions with tensile properties. The concentration of sulfur at grain boundaries was shown to govern the extent of intergranular fracture with increasing sulfur concentration resulting in increasing embrittlement. In the case of hydrogen, detection on an atomic scale is problematic; consequently, no direct quantitative correlation has been made between hydrogen segregation at grain boundaries and embrittlement. However, some studies of hydrogen embrittlement of nickel suggest that grain boundary segregation may play an important role in embrittlement.

The concentration of hydrogen at grain boundaries, caused by diffusion and subsequent trapping in a closed system, will be controlled by the thermodynamics of hydrogen segregation and the kinetics of hydrogen transport in the lattice. Hence, the thermal history of the nickel-hydrogen alloy will play a key role in segregation. Assuming that there is a negative binding enthalpy between hydrogen and grain boundary trapping sites, as the thermal energy of the material is lowered, a greater percentage of trap sites will be filled at equilibrium (provided they are not saturated). However, at the same time the flux of hydrogen to trapping sites will decrease because of the positive activation energy for diffusion (8). Direct observation of diffusive hydrogen segregation in nickel, as described above, has been made using secondary ion mass spectroscopy (SIMS) techniques (9). Measurements indicated that a significant amount of hydrogen segregation can occur at both grain boundaries and on free surfaces.

The present study focuses on the role of diffusive segregation of hydrogen before deformation on the embrittlement of nickel. Thermally charged tensile specimens were aged at several temperatures for various periods of time to allow for diffusion of hydrogen. The material was then quenched and tested in liquid nitrogen (77 K) so that hydrogen distribution obtained by aging would be essentially unaltered. This test method allowed the effect of diffusive segregation on the intergranular fracture mode to be isolated. Assuming that a unique relationship exists between grain boundary hydrogen segregation and intergranular fracture, the change in fracture mode caused by the various aging treatments is employed to study the thermodynamics and kinetics of hydrogen segregation in nickel.

### Experimental Procedure

Flat notched tensile specimens 6.0 mm wide, 0.66 mm thick, and with a gauge length of 20 mm were fabricated from cold rolled, high-purity nickel sheet. The total chemical impurity content of the test material was determined to be less than 100 ppm. After machining, the specimens were annealed in a  $N_2$ -5% $H_2$  atmosphere for 1 hour at 1573 K which resulted in an average grain diameter of approximately 800  $\mu m$ . Hydrogen was introduced into the specimens by heating to 1425 K for approximately 3 minutes in a static hydrogen-argon gas atmosphere followed by a rapid quench into water at room temperature without removal from the atmosphere. This charging procedure is believed to produce a relatively uniform distribution of hydrogen throughout the specimen. The desired solute hydrogen content was obtained by adjusting the partial pressure of hydrogen between 0.0 and 101 kPa prior to heating. The resulting solute hydrogen concentrations were calculated using Sievert's law and the solubility data of Robertson (8).

After quenching into water, specimens were held at ambient temperature for approximately 30 seconds to allow the furnace atmosphere to be exchanged with an inert gas, after which they were stored in liquid nitrogen. Prior to testing, samples were aged in temperature controlled methanol baths to allow diffusion of hydrogen. Three temperatures were used to age samples: 318 K, 253 K, and 208 K. Aging periods varied between 10 seconds to approximately  $10^7$  seconds.

Because hydrogen is essentially immobile in nickel at the temperature of liquid nitrogen, 77 K, tensile testing was performed in a liquid nitrogen bath to maintain hydrogen distributions obtained by aging. Prior to testing, specimens were mounted in a tensile stage that was immersed in liquid nitrogen, and were exposed to an ambient temperature only for brief moments while they were being transferred from one nitrogen bath to the next. Testing was performed at a nominal strain rate of  $3.3 \times 10^{-4} S^{-1}$ .

### Experimental Results

The fracture modes of tested samples were found to be either ductile shear rupture, intergranular, or some combination of these modes. Examples of these fracture modes are shown in Fig. 1. The percentage of intergranular fracture, determined by a linear intercept method, was used as a measure of embrittlement. Experimental results are plotted in the form of percent intergranular fracture versus aging time or bulk hydrogen concentration.

#### Fracture Mode Dependence on the Aging Period

To investigate the effect of aging time on embrittlement, individual specimens from sets of specimens with constant bulk hydrogen concentrations, were aged for various periods of time prior to testing. It was found that for a given aging temperature, only a certain range of solute hydrogen concentration will produce intermediate amounts of grain boundary fracture. Because of this, the bulk hydrogen concentration was adjusted

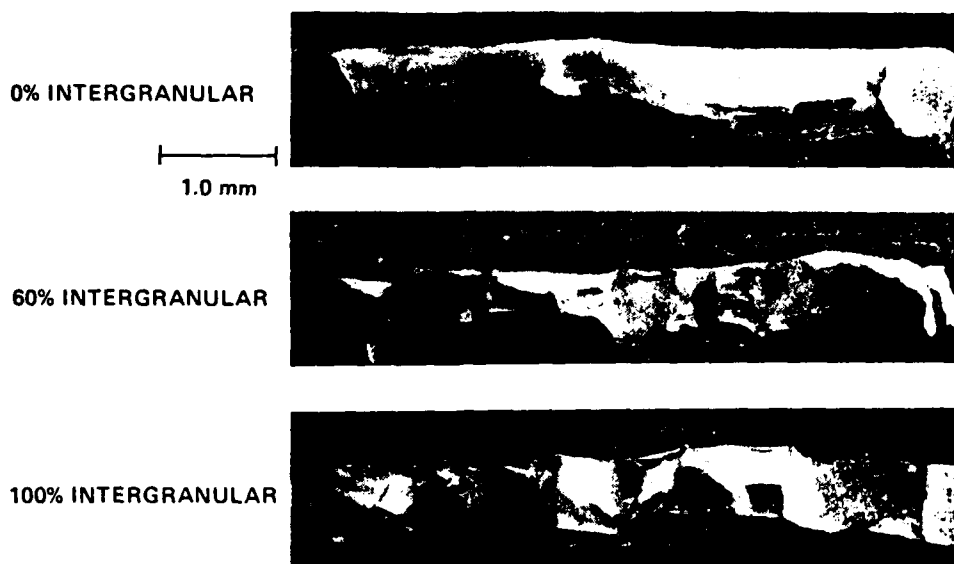


Figure 1 - SEM fractographs of typical fracture surfaces.

for some sets of specimens so that the maximum amount of intergranular fracture was somewhat less than 100%. By doing this, the change in fracture mode as a function of aging time can be used to investigate the kinetics of hydrogen segregation to grain boundaries.

Because the specimens were highly supersaturated with hydrogen at all times subsequent to charging, some, although relatively negligible, loss of hydrogen caused by outgassing occurred (except where noted). Because of this, the condition which maximizes embrittlement is not true equilibrium and will be referred to as "quasi-equilibrium." The aging time at the onset of maximum embrittlement (quasi-equilibrium condition) is denoted the "critical aging time."

The results of aging samples containing 95 ppm and 45 ppm hydrogen at 208 K are given in Fig. 2. The fracture mode of specimens containing 95 ppm hydrogen changes from zero to 100% intergranular fracture after aging for a period of approximately  $10^6$  seconds. Samples containing 45 ppm hydrogen reach a maximum percentage of intergranular fracture equal to approximately 25%. Similar results for specimens containing 275 ppm, 440 ppm, and 660 ppm of hydrogen which are aged at 253 K are shown in Fig. 3.

Figure 4 shows the effects of aging time at 318 K for specimens containing 765 ppm of hydrogen. As the aging time increases, the degree of intergranular fracture increases from about 30% at 10 seconds to 70% between 200 and  $10^4$  seconds. Upon further aging, the amount of intergranular fracture decreases because of the loss of hydrogen from outgassing. (This conclusion is based on comparison of the calculated RMS diffusion distance of hydrogen at 318 K during long aging periods with the width of the specimen.)

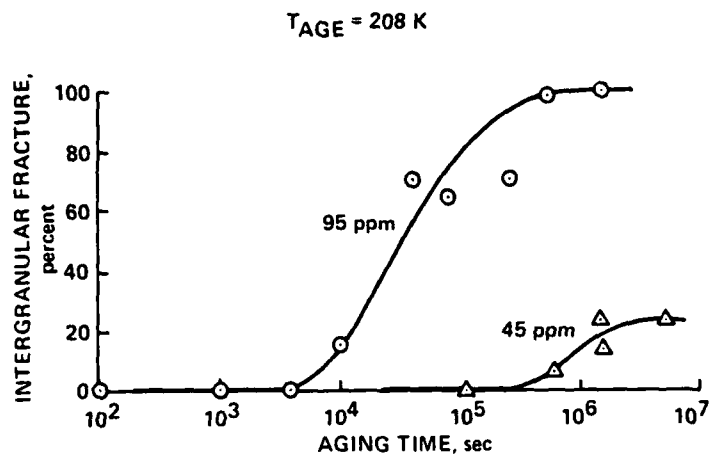


Figure 2 - Variation of the amount of intergranular fracture with aging time at 208 K for specimens containing 95 and 45 ppm H. The critical aging time, as defined in the text, is  $2.0 \times 10^6$  seconds.

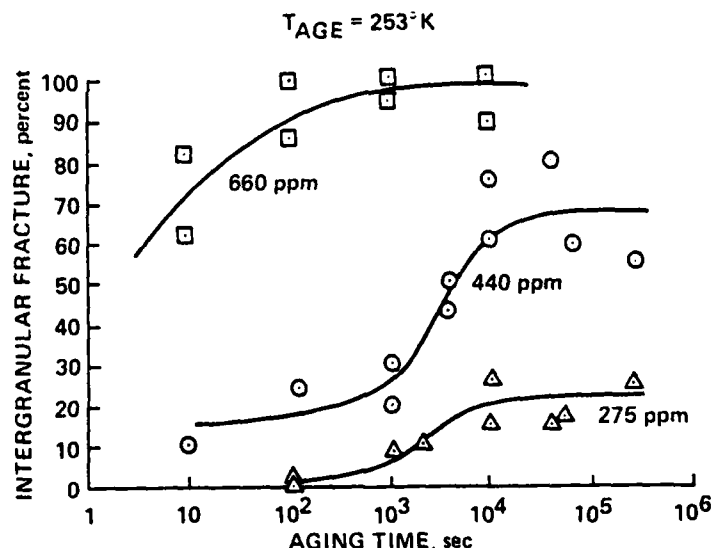


Figure 3 - Variation of the amount of intergranular fracture with aging time at 253 K for specimens containing 660, 440, and 275 ppm H. The critical aging time is  $1.0 \times 10^4$  seconds.



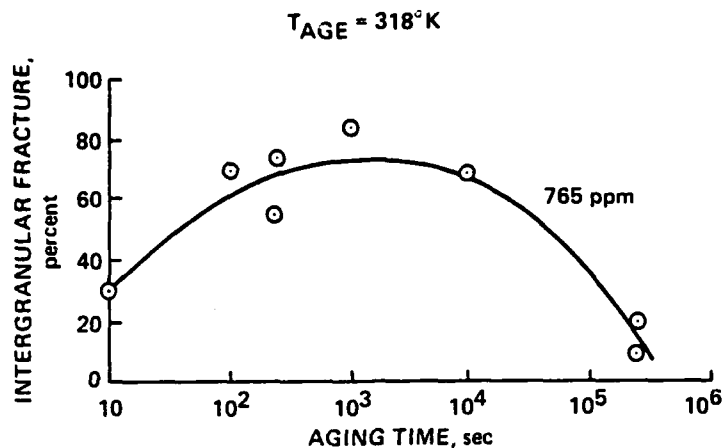


Figure 4 - Variation of the amount of intergranular fracture with aging time at 318 K for specimens containing 765 ppm H. The critical aging time is less than or equal to  $2.4 \times 10^2$  seconds.

The above data indicate that if hydrogen is allowed to diffuse prior to testing, significant increases in the amount of intergranular fracture can occur, particularly at the lower aging temperatures. As the aging temperature is decreased, longer aging periods are required to reach the maximum amount of intergranular fracture. Quasi-equilibrium occurs at "critical aging times" of  $2.0 \times 10^6$  and  $1.0 \times 10^4$  seconds for aging temperatures of 208 K and 253 K, respectively. The plot of intergranular fracture versus aging time for the 318 K aging temperature (Fig. 4) does not clearly indicate a critical aging time; however, it may be estimated to be less than, or equal to, 240 seconds.

#### Fracture Mode Dependence on Bulk Hydrogen Concentration

Specimens aged for a period of time appropriate to achieve quasi-equilibrium at a particular aging temperature exhibited a transition from ductile to intergranular fracture as the bulk hydrogen concentration was increased, as shown in Fig. 5 for the three aging temperatures. Solid points represent the average maximum values of intergranular fracture attained from aging studies (Figs. 2, 3, and 4). As an example, the average maximum value of intergranular fracture for specimens with 440 ppm hydrogen aged at 253 K is 70% (Fig. 3). This value was plotted (solid point) as part of the 253 K curve in Fig. 5 at the 440 ppm hydrogen composition. The open points in Fig. 5 represent the results of single tests obtained at the indicated compositions after aging the specimens for long enough time periods so as to attain quasi-equilibrium.

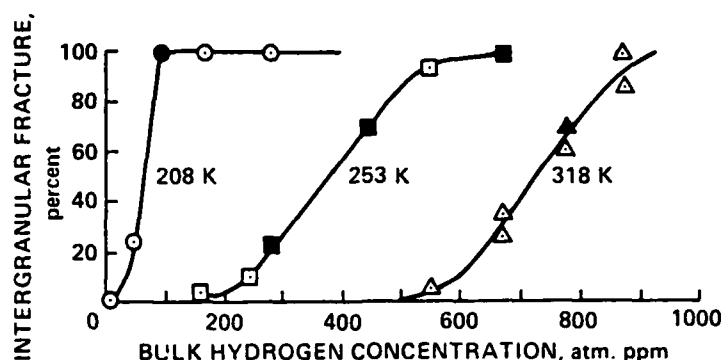


Figure 5 - Variation of the amount of intergranular fracture with bulk hydrogen concentration for specimens aged to achieved quasi-equilibrium. Open symbols denote specimens which were aged for times equal to  $t_c$ . Solid symbols denote the levels of intergranular fracture taken from Figs. 2-4.

#### Analysis and Discussion of Results

The data presented in this work clearly show that intergranular fracture of hydrogen charged nickel deformed at 77 K is strongly dependent on bulk hydrogen concentration and the thermal history of the material (aging temperature and time) prior to testing. These parameters are assumed to control the segregated grain boundary hydrogen concentration, which in the following analyses is assumed to have a solitary influence on the probability of intergranular fracture. It has been suggested that hydrogen can have an effect on the general deformation of charged nickel which may promote grain boundary fracture (5). At the test temperature of 77 K, hydrogen is essentially immobile and is not expected to have an effect on the general deformation behavior of nickel. To confirm this, the dislocation structure of hydrogen-charged and hydrogen-free specimens deformed at 77 K to 35% elongation were studied using transmission electron microscopy (TEM). There was no indication that hydrogen had any effect on the resulting dislocation character, density, or cell size. Thus it is not expected that the levels of bulk hydrogen concentration used in this study had any effect on intergranular fracture other than to serve as a source of hydrogen for segregation to grain boundaries, i.e., a unique relationship exists between the extent of grain boundary segregation of hydrogen and the amount of intergranular fracture observed.

#### Thermodynamics of Hydrogen Segregation

The dependence of the fracture mode on the bulk hydrogen concentration and aging temperature under quasi-equilibrium conditions (Fig. 5) can be used to analyze the thermodynamics of hydrogen segregation to grain boundaries. An expression for the equilibrium concentration of solute at a

grain boundary has been derived by McLean (10) using statistical mechanics and is given in Eqn. 1,

$$\frac{C_B}{1 - C_B} = \frac{C_0}{1 - C_0} \exp \frac{-H_B}{kT_{age}} \quad (1)$$

where  $C_B$  is the fraction of the grain boundary sites which contain H-solutes,  $C_0$  is the fraction of H-solute atoms in the bulk,  $k$  is the Boltzmann's constant,  $T_{age}$  is the equilibration temperature, and  $H_B$  is the binding enthalpy of the hydrogen to the grain boundary trapping sites. Equation 1 is essentially an expression of Fermi-Dirac statistics.

From Fig. 5 it can be determined that at the aging, or equilibration, temperatures of 208 K, 253 K, and 318 K the bulk hydrogen concentrations,  $C_0$ , required to promote 50% intergranular fracture are 70 ppm, 370 ppm, and 720 ppm, respectively. Assuming that 50% intergranular fracture corresponds to a single value of grain boundary hydrogen concentration,  $C_B$ , then a least-squares fit of Eqn. 1 to the data yields an enthalpy of segregation,  $H_B$ , equal to -11.6 kJ/mol (-0.12 eV) with no significant variation in the values obtained using data at other levels of intergranular fracture. This value of the binding enthalpy of hydrogen to grain boundaries compares favorably with binding enthalpies of hydrogen to point defects (11) and dislocations (1) in nickel which are generally on the order of 10 kJ/mol.

Using the value of 11.6 kJ/mol for  $H_B$ , Eqn. 1 is plotted for the three aging temperatures used in this study in Fig. 6. An approximated

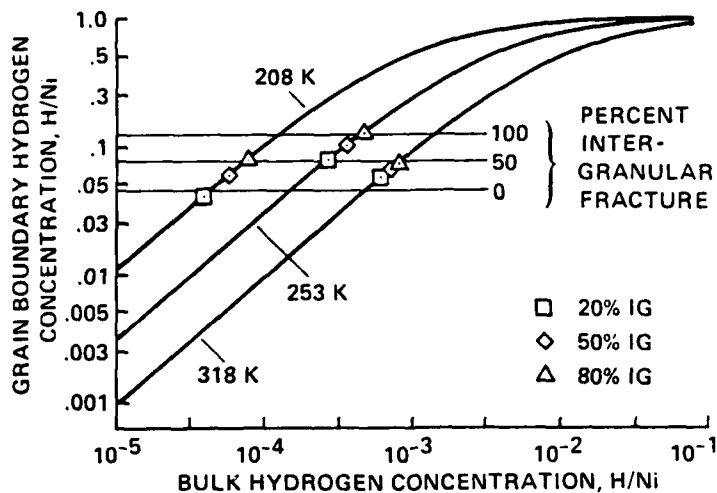


Figure 6 - Grain boundary hydrogen concentration plotted versus bulk hydrogen concentration (Eqn. 1,  $H_B = 11.6$  kJ/mol) for equilibration temperatures 208 K, 253 K, and 318 K. The bulk hydrogen concentrations corresponding to 20%, 50%, and 80% intergranular fracture for the three aging temperatures (taken from Fig. 5) are plotted on the isotherms. The right-hand scale relates intergranular fracture with grain boundary hydrogen concentration. The transition between ductile rupture and 100% intergranular fracture occurs approximately over the range of grain boundary hydrogen concentrations 5% to 15%.

percent intergranular fracture scale in the same figure indicates grain boundary nickel-hydrogen compositions corresponding to the transition between ductile and brittle fracture. From this figure, the ductile to brittle transition occurs over grain boundary hydrogen concentrations of approximately 0.05 to 0.15.

#### Kinetics of Hydrogen Segregation

An expression for the rate at which the grain boundary solute concentration increases during aging has been derived by McLean (10) using classical diffusion theory and is as follows:

$$\frac{C_{Bt} - C_{B'}}{C_B - C_{B'}} = 1 - \exp\left[-\frac{4Dt}{\alpha d^2}\right] \operatorname{erfc}\left[\frac{2(Dt)^{1/2}}{\alpha d}\right] \quad (2)$$

where

$C_{Bt}$  is the grain boundary concentration after time  $t$  at the aging temperature,

$C_{B'}$  is the initial grain boundary concentration,

$C_B$  is the equilibrium grain boundary concentration,

$\alpha$  is approximated by  $\exp(-H_B/kT)$ ,

$t$  is the aging time,

$d$  is the effective grain boundary thickness over which hydrogen segregation occurs,

$D$  is the diffusivity of H in nickel,

and

$$\operatorname{erfc}(x) = 1 - \operatorname{erf}(x)$$

In this treatment the grain boundary is modeled as a volume of constant solute concentration which increases with time by diffusion from the bulk. In the following section Eqn. 2 is fitted to intergranular fracture versus aging period data to determine the effective grain boundary thickness,  $d$ . Equation 2 is also used to examine the dependence of the critical aging times on aging temperature.

Effective Grain Boundary Thickness. The left-hand side of Eqn. 2, normalized grain boundary hydrogen concentration, is plotted versus aging time in Fig. 7 for the aging temperature 253 K using values of grain boundary thickness ranging from 30 Å to 3000 Å. Superposition of normalized intergranular fracture versus aging time for this temperature (440 ppm curve, Fig. 2) indicates that grain boundary hydrogen concentration correlates well with intergranular fracture if the grain boundary is modeled as

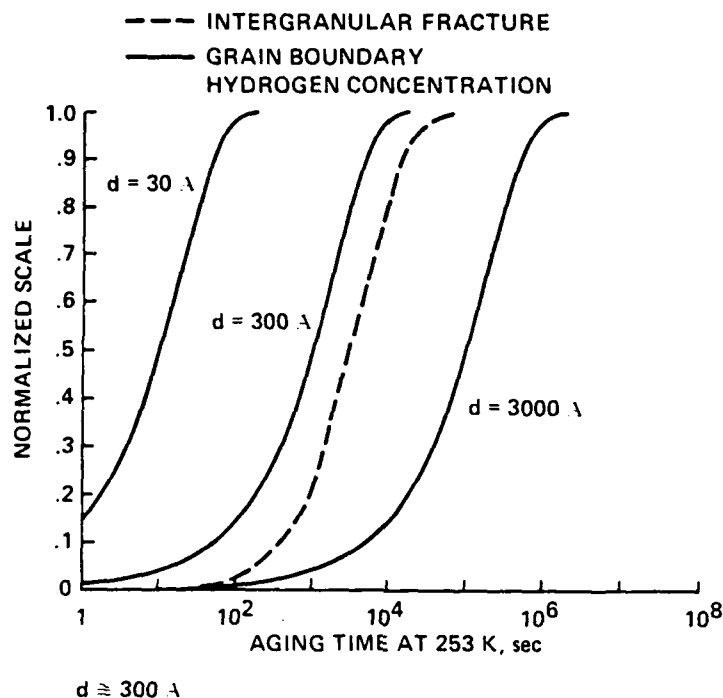


Figure 7 - Normalized grain boundary hydrogen concentration and intergranular fracture as functions of aging time at 253 K. Both grain boundary hydrogen concentration and intergranular fracture reach their maximum values after approximately the same aging period if the grain boundary is modeled as a 300 Å-thick region.

a 300 Å-thick region. A similar result is produced by superimposing normalized intergranular fracture on a plot of normalized grain boundary concentration versus aging time for the aging temperature 208 K. This analysis suggests that a relatively thick layer of approximately 300 Å of hydrogen enrichment occurs at grain boundaries, in agreement with the SIMS observations of Fukushima and Birnbaum (7). In contrast, the depth of equilibrium segregation of impurity elements such as sulfur and phosphorus at nickel grain boundaries has been shown by AES analysis to extend for a distance on the order of a monolayer (12). This difference suggests that the detailed thermodynamics of hydrogen segregation of hydrogen in nickel are different than in those of larger, less mobile impurity elements.

Critical Aging Time-Dependence on Aging Temperature. The critical aging time represents the period of time required to maximize hydrogen segregation at grain boundaries. Assuming that the effective grain boundary thickness over which hydrogen segregation occurs,  $d$ , is a constant (i.e., not a function of aging temperature), from Eqn. 2 the critical aging time dependence on aging temperature can be expressed as:

$$t_c = A \exp \frac{Q - 2H_B}{kT} \quad (3)$$

where  $Q$  is the activation energy for hydrogen diffusion in nickel and  $A$  is a constant. From this expression the natural logarithm of the critical aging time should be proportional to the inverse of the aging temperature, with the proportionality constant equal to  $(Q - 2H_B)/R$ . Using the value of  $H_B$  determined in this work (0.12 eV) and the value of  $Q$  from Reference 8, the critical aging times at 208 K and 253 K are shown in Fig. 8 to be in good agreement with this slope. No judgment can be made with respect to the critical aging time at 318 K since this was quoted as a maximum value and is quite possibly much less.

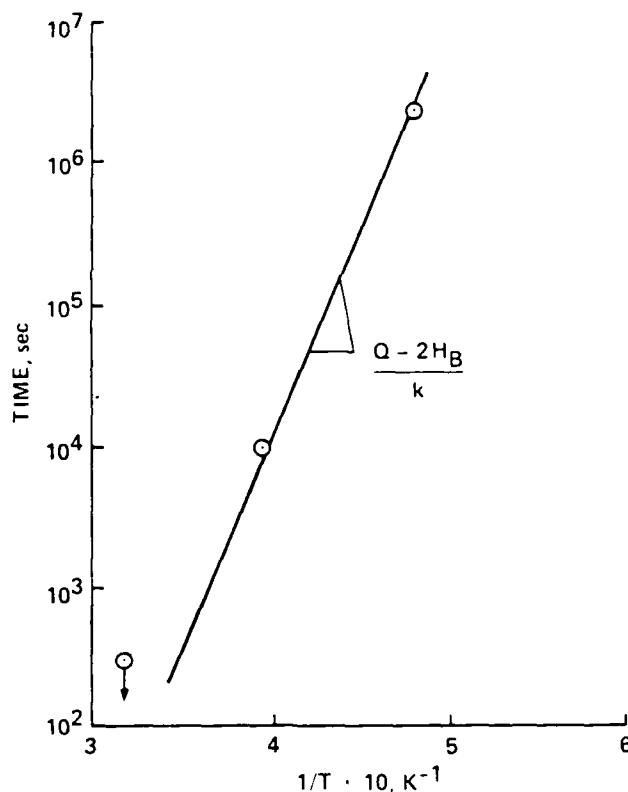


Figure 8 - Arrhenius temperature dependence of the critical aging time,  $t_c$ . The slope shown,  $(Q - 2H_B)/R$ , is the theoretical dependence of  $t_c$  on the inverse of the aging temperature.

### Conclusions

Embrittlement of hydrogen-charged nickel under test conditions where hydrogen is essentially immobile has been shown to be a function of bulk hydrogen concentration, and the thermal history of the nickel-hydrogen alloy prior to testing. These parameters are believed to control the concentration of hydrogen segregated at grain boundaries which, in turn, is assumed to govern intergranular fracture. The transition between ductile and intergranular fracture as a function of bulk hydrogen concentration and aging parameters was used to investigate the thermodynamics and kinetics of segregation. Analysis of the test data indicates that the equilibrium concentration of hydrogen at grain boundary trapping sites can be expressed in terms of Fermi-Dirac statistics and that the problem of ascertaining the concentration dependence on aging time can be modeled using classical diffusion theory. Conclusions from the analytical modeling performed in this work are as follows:

1. The binding enthalpy of hydrogen to grain boundary trapping sites is 11.6 kJ/mol (0.12 eV).
2. The transition from ductile to 100% intergranular fracture occurs approximately over the range of grain boundary hydrogen concentration from 5% to 15%.
3. A layer, on the order of 300 Å, of hydrogen enrichment occurs at nickel grain boundaries.

### Acknowledgments

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